



(51) International Patent Classification:
C08J 11/08 (2006.01)

(21) International Application Number:
PCT/IB2024/055790

(22) International Filing Date:
13 June 2024 (13.06.2024)

(25) Filing Language: Italian

(26) Publication Language: English

(30) Priority Data:
102023000012114 13 June 2023 (13.06.2023) IT

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CV, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IQ, IR, IS, IT, JM, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, MG, MK, MN, MU, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, CV, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SC, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, ME, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

(54) Title: POLYAMIDE RECOVERY PROCESS

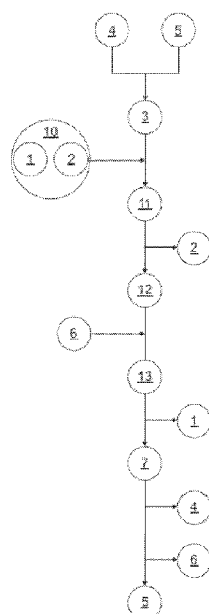


Fig. 1

(57) Abstract: Provided is a process for recovery of polyamides (1) from a material (10) comprising polyamides (1) and pollutants (2), said process comprising: a phase of preparing a first solution (3) being a deep eutectic solvent and including at least methanol (4) and at least one salt (5); a solubilization phase in which the material (10) is added to the first solution (3) to obtain a first mixture (11) in which the polyamides (1) bind to the salt (5); a first filtration phase in which pollutants (2) are separated from the first mixture (11) to obtain a second mixture (12) including polyamides (1) bound to salt (5) and methanol (4); a separation phase in which a separating agent (6) is added to the second mixture (12) to subtract the salt (5) from both the methanol (4) and the polyamides (1) and obtain a third mixture (13) in which the salt (5) and the polyamides (1) are again separated; a second filtration phase in which the third mixture (13) is subjected to filtration so as to obtain the polyamides (1) and a second solution (7).

Published:

- *with international search report (Art. 21(3))*
- *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))*
- *in black and white; the international application as filed contained color or greyscale and is available for download from PATENTSCOPE*

POLYAMIDE RECOVERY PROCESS

The object of the present invention is a polyamide recovery process of the type specified in the preamble of the first claim.

The object of the present invention is a polyamide recovery process that finds application primarily, but not exclusively, in the chemical industry and, in particular, in materials recycling.

Techniques for recovering polyamides from waste materials are currently known. One type of approach to the recovery of polyamides is the one based on depolymerization. This type of recovery is mainly used for polymers obtained by "stage growth" polymerization, such as polyesters, polycarbonates, polyamides (PA), etc. The disadvantage of this technique is that it allows the monomer or oligomers of the starting polymer to be obtained with a high degree of impurities present in the resulting mixture. The presence of impurities makes it problematic to reuse the monomer to carry out a new polymerization, since a high purity of the monomer is required, which is difficult to obtain for materials from waste.

For this reason, more effective techniques for the recovery of polyamides are being sought. In detail, these techniques are based on the separation and chemical purification of the polymer. The separation of the polymer from other polymers and materials to which the former is often bonded is complex. As a matter of fact, these processes require use of different solvents depending on the components to be separated and filtration stages. Typically, a chemical separation and purification process includes three main phases: in the first phase the material containing the polymer to be separated is dissolved in a solvent in which the polymer is soluble. In this phase it is required to select a solvent having specific affinity for the polymer to be separated and not for pollutants or other bound polymers. This produces a solution of the dissolved polymer in the affine solvent. In the second phase filtration of the polymer solution is implemented so as to separate undissolved pollutants.

Finally, a stage is implemented aimed at recovering the polymer of interest from the solution by separating it from the solvent. The latter stage may, for example, be implemented via evaporation of the solvent.

Solvents such as propylene glycol, alkenic carbonates and Dimethyl sulfoxide (DMSO) are known to be used.

The prior art implies some important drawbacks.

In particular, the drawback of using these solvents for PA recovery is related to the need to use high process temperatures, both to solubilize the PA of interest and to reduce the viscosity of the solution so that the latter is more easily filtered.

Recovery of known solvents also involves high-temperature processes because of the high boiling points of the solvents.

Finally, high temperatures may cause thermal shocks to the polymer.

CN 114 800 943 A describes a method for recycling waste mats.

In this situation, the technical task underlying the present invention is to devise a process for recovering polyamides that may substantially obviate at least some of the mentioned drawbacks.

Within said technical task it is an important object of the invention to obtain a process that requires low process temperatures.

Another important task of the invention is to allow for a polyamide recovery process easier to implement.

Finally, another important object of the invention is to allow for a polyamide recovery process having a reduced environmental impact.

The specified technical task and object are achieved by a polyamide recovery process as claimed in the appended claim 1.

Preferred technical solutions are highlighted in the dependent claims.

The characteristics and advantages of the invention are clarified below by the detailed description of preferred embodiments of the invention with reference to the appended drawings, wherein:

Fig. 1 shows a diagram of the process according to the invention, and

Fig. 2 illustrates an alternative diagram of the process according to the invention.

In this document, measurements, values, shapes, and geometric references (such as perpendicularity and parallelism), when associated with words such as "approximately" or other similar terms such as "nearly" or "substantially," may be affected by production and/or manufacturing measurement errors or inaccuracies and, most importantly, may be affected by a slight deviation from the value, measurement, shape, or geometric reference with which it is associated. For example, when associated with a given value, such terms, preferably indicate a deviation of no more than 10 percent of that value.

Also, when used, terms such as "first," "second," "top," "bottom," "main," and "secondary" do not necessarily identify an order, priority of relationship, or relative position, but may simply be used to more clearly distinguish different components from each other.

Unless otherwise specified, as reflected in the following discussions, terms such as "processing," "computing," "determination," "computation," or the like are considered to refer to the action and/or processes of a computer or similar electronic computing device that manipulates and/or transforms data represented as physical, such as electronic quantities of records of a computer system and/or memories in other data similarly represented as physical quantities within computer systems, registers, or other information storage, transmission, or display devices.

Unless otherwise indicated, the measurements and data herein indicated are to be considered as having been taken according to ICAO International Standard Atmosphere (ISO 2533:1975).

With reference to the Figures, the polyamides subject to the recovery process according to the invention are collectively indicated by the reference number 1.

Polyamides 1 are a class of polymers in which the monomeric units constituting the polymer chain are linked together by amide bonds. They have the advantage of high tensile and frictional strength. Therefore, they are used, either as the only material or in combination with other polymers in the manufacturing of products for which high technical characteristics and resistance to environmental factors are required. In particular, polyamides 1 may be used in the production of fibers, fabrics, nets, cordage, gaskets, etc. In addition, they may be processed by the usual techniques used for thermoplastic polymers, such as extrusion, injection molding, blow molding, etc.

The invention comprises a novel process for recovering polyamides 1 starting from a material 10. The material 10 is preferably a waste material, such as post-consumer fabric or cordage artifacts, or fishing nets, airbag balls,

fabric floor coverings, packaging sheets or films, or a waste from industrial production of said artifacts. Thus, material 10 includes polyamides 1 and pollutants 2. Pollutants 2 include materials of various kinds depending on the original use for which material 10 was intended before use. For example, pollutants 2 may include fabrics containing fibers of various kinds, such as, for example, carbon fibers, polyolefin fibers, polyester fibers, elastomeric fibers, natural fibers, or fibers of other materials, woven with polyamide 1 fibers, for the production of clothing or work clothes such as gowns or overalls, etc., for example.

Material 10 may include pollutants 2 of another kind related to the use of material 10 itself. In this regard, cordage used for fishing nets may contain sand residues or shellfish shells clinging to the net.

Pollutants 2 may also be coating films or sealing agents. For example, pollutants 2 may consist in silicone resins used to waterproof the polyamide 1 fabric of airbag inflators in order to thereby prevent the escape of gases in the event of an accident.

The recovery process preferably includes a preliminary phase of material 10 size reduction. Size reduction has the advantage of making material 10 more easily processable. For example, if material 10 is a fabric, size reduction may be implemented via cutting or fraying operations. Due to size reduction, the recovery process is therefore rendered more efficient and quicker.

In the process, polyamides 1 preferably include a polymer to be selected between nylon 6 and nylon 6,6. This type of polymer is the one most widely used in the applications previously described. It is also effectively recovered by the process according to the invention.

The process includes a phase of preparing a first solution 3.

The first solution 3 is advantageously a deep eutectic solvent. As a matter of fact, it allows polyamides 1 to be solubilized at lower temperatures than solvents used in common purification processes. Thus, polyamide 1 may advantageously be recovered by means of a process that is less energy-intensive than known purification processes. In addition, components typically used in deep eutectic solvents are less toxic than components utilized in solvents used in purifications.

Frequently, a deep eutectic solvent is formed when a solvent binds to a solute, such as a salt, by hydrogen bonds. Therefore, the function of the deep eutectic solvent solely consists in bringing the liquefied solute into contact with the solid material to be dissolved, which, in the case of the present invention, is solid polyamide. If the latter establishes hydrogen bonds with the solute in the deep eutectic solvent that are "stronger" than those between the solvent and the solute in the deep eutectic solvent, the solute will interact with the polyamide and modify it. In the case of polyamides, the modification produces a decrease in the attractive forces between the macromolecules of the polyamide resulting in the liquefaction of the same.

In this regard, solution 3 includes at least methanol 4 and at least one salt 5.

Methanol 4 is a solvent in the liquid state at polar room temperature and protic with a low boiling temperature. Therefore, its use is advantageous, since it also makes the temperatures required to produce a deep eutectic solvent capable of solubilizing polyamides 1 very low.

Salt 5 is one of the salts that cause deep eutectic solvent formation with methanol 4. In particular, it preferably includes at least one of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, NaSCN , $\text{CaI}_2 \cdot 4\text{H}_2\text{O}$, and calcium salicylate. This list of salts is

representative, but not exhaustive, of the salts that, when solubilized in methanol 4 to saturation, result in the formation of a deep eutectic solvent.

Once the first solution 3 has been prepared, the process includes a solubilization phase. In this phase, the material 10 is added to the first solution 3. The material 10 preferably already undergoes size reduction, as previously described.

In this way each monomeric unit of polyamide binds to salt 5 thus decreasing the forces of attraction between the macromolecules of polyamide 1, so polyamide 1 liquefies thereby becoming separable from solid pollutants 2.

In this way, a first mixture 11 is obtained. The first mixture 11 comprises material 10, methanol 4, salt 5 and liquefied polyamides 1. Since the amount of salt 5 bound to methanol 4 in the deep eutectic solvent obviously decreases at this stage, the amount of salt 5 is preferably kept constant in a 1:1 molar ratio with the monomeric residue of polyamides 1. Therefore, during the process further additions are implemented so that the molar ratio of Salt 5/Residue monomer of polyamide 1 is consistently about 1:1.

The next phase of the process includes an initial filtration phase in which the pollutants 2 are separated from the first mixture 11. Filtration may be implemented using a Buchner filter to separate the solid pollutants 2 from the liquid components.

The first filtration phase may advantageously be implemented at a temperature lower than the boiling temperature of each of the components of the deep eutectic solvent, i.e. lower than the boiling temperature of methanol 4. In this way, the energy consumption required to separate the polyamides 1 from the pollutants 2 is advantageously reduced with a consequent reduction in process costs. In this way a second mixture 12 is obtained. It includes the polyamides 1 linked to salt 5 and methanol 4.

Subsequently, the process includes a separation phase in which a separating agent 6 is added to the second mixture 12. The separating agent 6 is a substance having the function of removing the salt 5 from both the methanol 4 and the polyamides 1. In this way, a third mixture 13 is obtained. It deals with a mixture containing methanol 4, salt 5, polyamides 1 as well as the separating agent 6 bound to the salt 5. The separating agent 6 performs the function of breaking the bond between salt 5 and polyamides 1. In particular, the separating agent 6 includes a large number of salt 5 solvents, including aliphatic alcohols in a number of carbon atoms bigger than methanol. In detail, the separating agent 6 preferably comprises at least one of the solvents selected from 1-butanol, acetamide and dimethyl sulfoxide. These liquid solvents have the advantage of being particularly effective in separating the salt 5 from the polyamides 1. In particular, they provide benefits where the polyamides 1 include nylon 6 or nylon 6,6. At this stage, polyamides may precipitate as a solid into powder.

Therefore, in the third mixture 13 the salt 5 and the polyamides 1 are separated again.

After the separation phase, a second filtration phase follows. In this phase, the third mixture 13 is subjected to filtration in order to obtain the polyamides 1 and a second solution 7. The latter includes methanol 4, salt 5 and the separating agent 6. At this stage, the polyamides 1 are separated and purified. The second filtration phase may also be implemented by using a Buchner filter to separate the solid polyamides 1 from the second solution 7. This second filtration phase provides benefits since it takes place at a temperature lower than the boiling temperature of methanol 4. As already mentioned, the filtration and purification process provides benefits in terms

of energy and cost.

The process preferably includes a recovery phase following the second filtration phase. In the recovery phase, methanol 4, salt 5 and the separating agent 6 are separated from the second solution 7. These separation phases may be implemented by distillation from the most volatile to the least volatile component. For example, the distillation of methanol 4 may be implemented first, which has a lower boiling temperature than that of the other components present in the second solution 7. Next, distillation of the separating agent 6 is carried out occurs. At the end of the recovery phase, methanol 4, salt 5 and separating agent 6 are re-obtained separately. This additional phase, once the polyamides 1 are separated, has the advantage of recovering the substances used during the process with process costs being reduced. In addition, the process is more environmentally friendly, since the solvents and salts used may be recovered, which are not released into the environment.

In the process the methanol 4 is preferably separated from the second mixture 12 before the filtration of the polyamides 1. Therefore, in some embodiments of the process implementation, the phase of achieving polyamides 1 purification may follow a different order.

In detail, following the first filtration phase, the second mixture 12 preferably is subjected to a first partial separation phase in which methanol 4 is separated from the second mixture 12 via distillation. In this way, a fourth mixture 14 is obtained. It includes salt 5 and polyamides 1. This mixture is in a solid state and, in particular, may be in powder form.

The fourth mixture 14 is subjected to a second partial recovery phase in which the separating agent 6 is added to the former. In this way, a fifth mixture 15 is obtained. The addition of liquid separating agent 6 may cause salt 5 to be solvated wherein only polyamides 1 remains in a solid state.

The second partial recovery phase is followed by the second filtration phase, in which the fifth mixture 15 is subjected to filtration in order to separate polyamides 1 and obtain a third solution 8. The third solution 8 is a solution of salt 5 in the separating agent 6.

At this stage in the process, polyamides 1 are separated and recovered.

Finally, in the recovery phase, the separating agent 6 is separated from the third solution 8 so as to obtain salt 5 as well as the separating agent 6.

In an exemplary implementation of the process, polyamides 1 comprises nylon 6 or nylon 6,6, salt 5 comprises NaSCN, and the separating agent 6 comprises 1-butanol. In detail, material 10 includes used airbags comprising nylon 6,6. In the preliminary size reduction phase, the airbags are frayed and shredded resulting in a frayed fabric.

In the phase of preparing the first solution 3 in a 500 cc flask, NaSCN is placed in an amount of methanol 4 preferably between 50 cc and 350 cc, more preferably between 100 cc and 300 cc, even more preferably between 150 cc and 250 cc. The NaSCN salt 5 is added until an initial solution 3 comprising methanol 4 saturated with NaSCN is obtained. In the next solubilization phase, material 10 in the form of frayed tissue is added to the first solution 3 under stirring at a temperature preferably below 64 °C, more preferably between 50 °C and 64 °C, even more preferably between 55 °C and 60 °C. In this way, the first mixture 11 is obtained. For example, an amount of frayed fabric may be added preferably between 100 g and 300 g, more preferably between 150 g and

250 g. Following the addition of material 10, one may continue to gradually add salt 5 so as to keep the first mixture 11 saturated. Additions are preferably made under the same temperature conditions as previously indicated. In this example, pollutants 2 comprise a silicone coating of nylon 6 fibers. After a certain time interval, the silicone coating separates from the nylon 6,6 fibers. At this point, the first filtration phase is conducted, in which the first mixture 11 is filtered through a Buchner filter to separate the solid pollutants 2. This phase is also preferably implemented while maintaining the temperature values of the previous phases. In this phase, the required temperature values have the advantage of avoiding solidification of the substances to be separated from the pollutants 2. Once the second mixture 12 is obtained, the first partial separation phase may be implemented in which the distillation of methanol 4 occurs. This phase may be implemented in a vacuum and under mechanical agitation. In this way, the precipitate may be pulverized. Once the methanol 4 is removed and the fourth solid 14 mixture is obtained, the second phase of partial recovery is carried out. In this phase, 1-butanol is added to the fourth 14 mixture. In detail, a volume of 1-butanol may be added preferably between 200 cc and 400 cc, more preferably between 250 cc and 350 cc. The addition may be done under stirring. Dissolution of NaSCN in 1-butanol is exothermic and causes heating of the fifth mixture 15 being formed. Once the fifth 15 mixture cools off, the second filtration phase is implemented. In this phase, filtration of the nylon 6,6, in pure solid powder is carried out, which is separated from the third saline solution 8 of 1-butanol. Once the recovery of pure nylon 6,6 occurred, the recovery phase may finally take place, in which the third solution 8 is subjected to vacuum distillation so as to separate the 1-butanol from the salt 5 NaSCN.

Other examples of the process for recovering nylon 6,6 from airbag scraps may occur in the same way as described, in which, instead of NaSCN, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{CaI}_2 \cdot 4\text{H}_2\text{O}$, or calcium salicylate may each be used as salt 5.

In another example of performing the process, material 10 comprises shredded sock production waste containing 97% nylon 6 fibers and 3% Lycra fibers. Thus polyamides 1 include nylon 6 fibers and pollutants 2 includes Lycra fibers. In this example, methanol 4 and NaSCN are used as in the previous examples. In the solubilization phase, material 10 is gradually added to a volume of the first solution 3 of NaSCN in methanol 4 preferably between 200 cc and 400 cc, more preferably between 250 cc and 350 cc. The amount of material 10 added is preferably between 100 g and 300 g, more preferably between 150 g and 250 g.

The process is conducted under the same conditions as the previously described examples. The differences from the described examples are shown below.

After the solubilization phase, the material 10 separates within the first mixture 11 so that the Lycra fibers are almost totally separated from the nylon 6 fibers, which are dispersed in methanol 4. In detail, nylon 6, unlike nylon 6,6, forms a dispersion of particles in methanol 4. The first filtration phase is implemented in the manner previously described to remove Lycra fibers, and all the following phases are carried out based on temperature ranges and amounts, which were previously indicated in the individual phases. Also in this example, 1-butanol is used as separating agent 6. At the end of the process, pure nylon 6 is obtained and the reagents used are recovered.

Other examples of a process for recovering nylon 6 from sock production waste may be carried out in the same

way as described, in which, instead of NaSCN, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{CaI}_2 \cdot 4\text{H}_2\text{O}$, or calcium salicylate may each be used as salt 5.

The invention includes a novel use of a deep eutectic solvent for the purification and recovery of polyamides. As previously described, through the use of deep eutectic solvents, polyamides may be recovered from production waste or residues at lower temperatures than known processes.

The process according to the invention achieves important advantages.

In fact, the use of deep eutectic solvents allows the process to be implemented at lower temperatures than those required in traditional purification processes, resulting in significant energy savings.

In addition, the lower temperatures make it possible to reduce solution viscosity and mixtures during the process as well as to avoid thermal shock to the polymer to be recovered.

Another advantage of the process is related to the fact that the mixtures used allow for total recovery of the substances used, making the process more environmentally friendly.

The environmental advantage is also, and more importantly, related to the fact that through this process, waste containing this type of polymer is prevented from being dispersed into the environment. In fact, these wastes that constitute material 10 cannot be arranged in landfills, since the heat balance of their combustion is negative. Therefore, if not recovered through recovery processes, they are released into the environment.

Moreover, the recovery process yields nylon powders that are particularly suitable for use in rotational molding processes. Thus, this process corresponds to both the recovery of nylon from residues and the production of rotational molding powders in a single process.

Finally, the process has the advantage of allowing for recovery and purification of nylon by means of a process easier to implement due to use of purification techniques for which suitable equipment already exists.

The invention is susceptible to variations within the scope of the inventive concept defined by the claims.

Within that scope all details are substitutable by equivalent elements and the materials, shapes and sizes can be of any kind.

CLAIMS

1. A process for recovering polyamides (1) from a material (10) comprising said polyamides (1) and pollutants (2), said process comprising:

- a phase of preparing an a first solution (3) comprising at least methanol (4) and at least one salt (5);
- 5 – a solubilization phase in which said material (10) is added to said first solution (3) to obtain a first mixture (11) in which said polyamides (1) bind to said salt (5);
- a first filtration phase in which said pollutants (2) are separated from said first mixture (11) to obtain a second mixture (12) including said polyamides (1) bound to said salt (5) and said methanol (4);
- a separation phase in which a separating agent (6) is added to said second mixture (12) to subtract said salt (5) from both said methanol (4) and said polyamides (1) and obtain a third mixture (13) in which said salt (5) and said polyamides (1) are again separated;
- 10 – a second filtration phase in which said third mixture (13) is subjected to filtration so as to obtain said polyamides (1) and a second solution (7);
- and characterized by the fact that
- 15 – said first solution (3) is a deep eutectic solvent.

2. Process according to claim 1, wherein said first filtration phase and/or said second filtration phase occurs at a temperature below the boiling temperature of said methanol (4).

3. Process according to claim 1 or 2, comprising a recovery phase, subsequent to said second filtration phase, wherein said methanol (4), said salt (5) and said separating agent (6) are separated from said second solution (7).

4. Process according to any one of the preceding claims, wherein said methanol (4) is separated from said second mixture (12) prior to said filtration of said polyamides (1).

5. Process according to the preceding claim wherein, subsequent to said first filtration phase, said second mixture (12) is subjected to a first partial separation phase in which said methanol (4) is separated from said second mixture (12) by distillation to obtain a fourth mixture (14).

6. Process according to the preceding claim, wherein said fourth mixture (14) is subjected to a second partial recovery phase wherein said fourth mixture (14) is added to said separating agent (6) so as to obtain a fifth mixture (15).

7. Process according to the preceding claim, wherein said second partial recovery phase is followed by said second filtration phase, wherein said fifth mixture (15) is subjected to filtration so as to separate said polyamides (1) and obtain a third solution (8).

8. Process according to the preceding claim, wherein, in said recovery phase, from said third solution (8) said separating agent (6) is separated in order that said salt (5) and said separating agent (6) are obtained separately.

9. Process according to any of the preceding claims, wherein said polyamides (1) include a polymer selected between nylon 6 and nylon 6,6.

10. Process according to any one of the preceding claims, wherein said salt (5) comprises at least one

selected from $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, NaSCN , $\text{CaI}_2 \cdot 4\text{H}_2\text{O}$ and calcium salicylate.

11. Process according to any one of the preceding claims, wherein said separating agent (6) comprises at least one selected from 1-butanol, acetamide and Dimethyl sulfoxide.

5 12. Process according to any one of the preceding claims, comprising a preliminary phase of reducing the size of said material (10).

13. Process according to any one of the preceding claims, wherein the amount of said salt (5) is maintained in a 1:1 molar ratio with the monomeric residue of said polyamides (1).

14. Use of a solution comprising at least methanol (4) and at least one salt (5), said solution being a deep eutectic solvent, for the purification and recovery of polyamides.

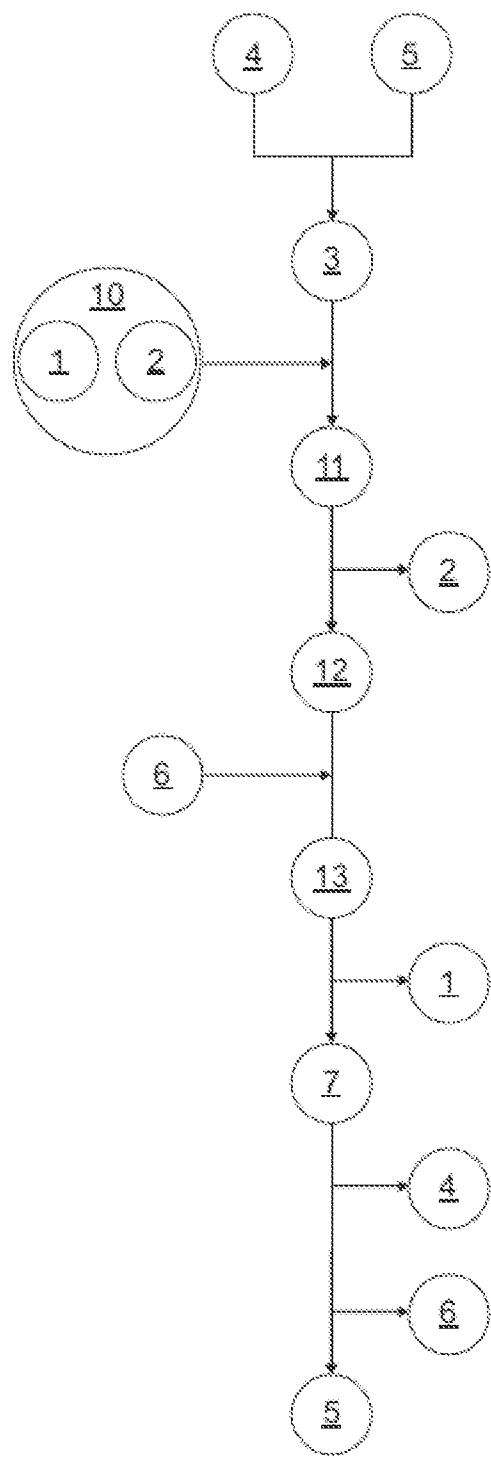


Fig. 1

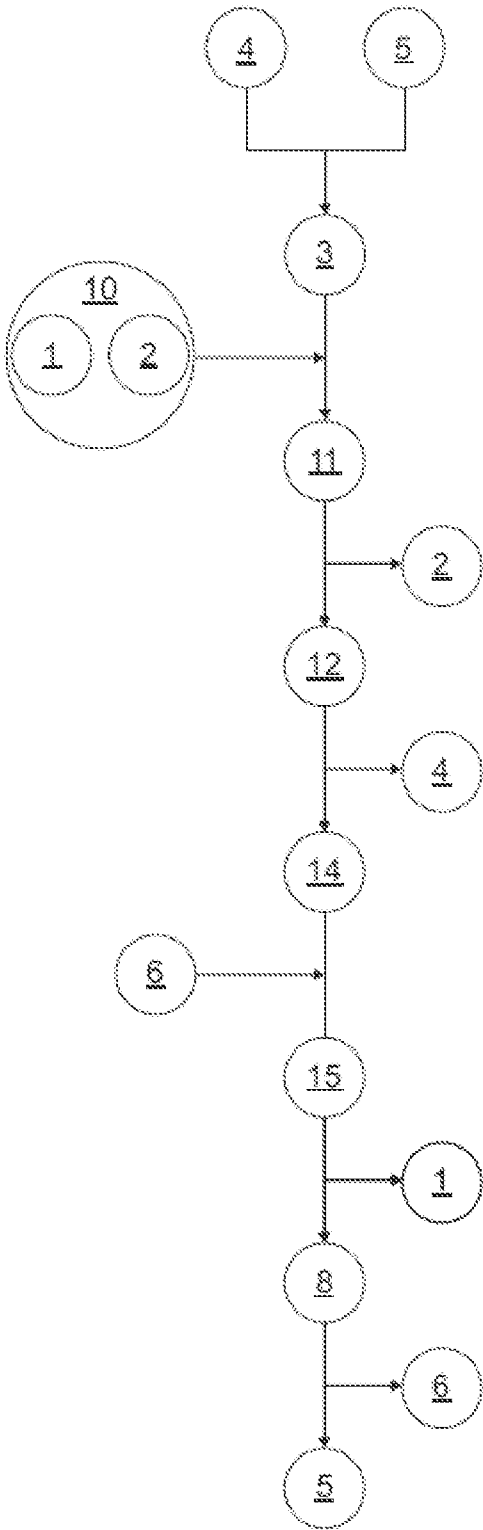


Fig. 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2024/055790

A. CLASSIFICATION OF SUBJECT MATTER

INV. C08J11/08

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 5 110704 B2 (UNIV KANSAI) 26 December 2012 (2012-12-26) no "DES"; figure 1; examples 2,4 -----	1 - 10, 12 - 14
A	CN 114 800 943 A (UNIV JIAXING) 29 July 2022 (2022-07-29) no methanol based "DES"; examples 1-3 -----	1 - 14
A	GB 563 078 A (DU PONT) 28 July 1944 (1944-07-28) examples I, II -----	1 - 14



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search

12 November 2024

Date of mailing of the international search report

26/11/2024

Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/IB2024/055790

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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